EVALUATING A CONTAMINATION HAZARD WITH A RESIDUAL GAS ANALYZER

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1.0 ABSTRACT

This report describes the use of a residual gas analyzer (RGA) to monitor the partial pressure of a suspect contaminant material during the bake-out of a vacuum chamber and also during the following test operation which involved a sensitive spacecraft instrument. A method for estimating the RGA sensitivity to the contaminant is presented. The method is considered to be conservative from the standpoint of assessing the contamination hazard arising from the material in question. An analysis of the test data is presented; it indicates that gross contamination did not occur. A cold finger technique that provides a surface on which residual gases can condense is also described. Data from the cold finger is used to infer that the sensitive test item had not been contaminated.

The techniques used are shown to be viable and methods for improving upon them are suggested.

2.0 BACKGROUND

In preparing for the environmental test of a sensitive ultraviolet telescope planned for use aboard a spacecraft, a significant quantity of DC-704* diffusion pump fluid was detected using cold finger techniques**. This was an unusual occurrence; the chamber previously had been used for highly sensitive experiments and careful analyses had rarely detected even trace quantities of this fluid.

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* A Dow Corning Corporation trade name; tetraphenyltetramethyltrisiloxane, molecular weight 484.

**Appendix A provides an explanation of the cold finger technique.

A cold finger test made six months before this test was scheduled showed less than 1 mg of residue with no DC-704 identified; a test conducted about one month earlier showed less than 1 mg of residue with no DC-704. However, a cold finger analysis conducted about three weeks before the telescope test was scheduled showed 76 mg of residue with DC-704 definitely identified. An analysis completed one week before the planned telescope test indicated 102 mg of residue and DC-704 appeared to be the major constituent.

A check of facility operating procedures uncovered a change that could result in the fluid being introduced into the test volume; the use of the new procedure was discontinued. However, the chamber was considered contaminated and unsuitable for conducting tests of sensitive items.

Because of schedules requiring that the test item undergo calibrations in the chamber at this point in time and other considerations, it was impossible to perform a thorough chamber cleaning; the need for fixturing, etc., made the use of other chambers impractical.

3.0 APPROACH

It was decided to conduct a short chamber bake-out and during the bake-out to monitor the level of DC-704 using an RGA. If one could assign a partial pressure to the RGA readings, then, knowing the vapor pressure characteristics of the DC-704, one could assess the hazard of its condensing on the sensitive surface of the test item. This would not preclude adsorption due to the surface forces but would provide an indication as to whether gross contamination would occur. The cold finger too could be used to assess chamber contamination.

A Granville Phillips Company, Spectra Scan 400 quadrupole RGA was used to monitor the partial pressure of the DC-704. A spectrum of DC-704 had been obtained with a similar spectrometer previously and a review of the spectrum, together with a review of previous chamber spectra, indicated that the peaks at mass to charge ratio (m/e) = 195 and 197 provided a good indicator for DC-704; they were significant in the DC-704 spectrum and were generally at a low level in scans made previously in various chambers in the laboratory.

Literature values for DC-704[1,2] also indicated that these mass numbers were appropriate. Peaks at m/e = 188 and 259 provided additional confirmation as to the identification of DC-704. Peaks above m/e = 300 were not usable because of the performance of the particular RGA; peaks below m/e = 188, while necessary to confirm the presence of DC-704, were present from many other sources. Peak height ratios among the selected peaks also confirmed the identification. The peak at m/e = 197 was chosen to be representative of the partial pressure of DC-704. Figure 1 is a portion of the RGA scan taken at time 91.7 hours during the test of the telescope.

Methods for establishing the relationship between the m/e = 197 peak height and the partial pressure of DC-704 were considered. A formal calibration of the RGA decided against because suitable facilities could not be made available in time. The use of literature values was considered but the corrections necessary to make them applicable to the particular RGA were considered subject to too many uncertainties. It was decided to use what was considered to be a conservative approach from the standpoint of contamination. The method simply assumed that the height of the m/e = 197 peak was equal to the saturation vapor pressure when the first reading, made as soon as possible after the beginning test, was taken. This pressure would be taken at the temperature of the chamber at that time (25°C). Two conservative factors were therefore involved. First, since the system was being dynamically pumped, the partial pressure would be less than the vapor pressure; by definition, the vapor pressure is an equilibrium value. Secondly, unless very large quantities of the oil existed in the chamber, the partial pressure could be expected to fall with time as the sources were depleted. Therefore, by the time the reading was taken, the partial pressure probably would have fallen by some amount.

Data on the vapor pressure of DC-704 was taken from the manufacturer's bulletin.[3]

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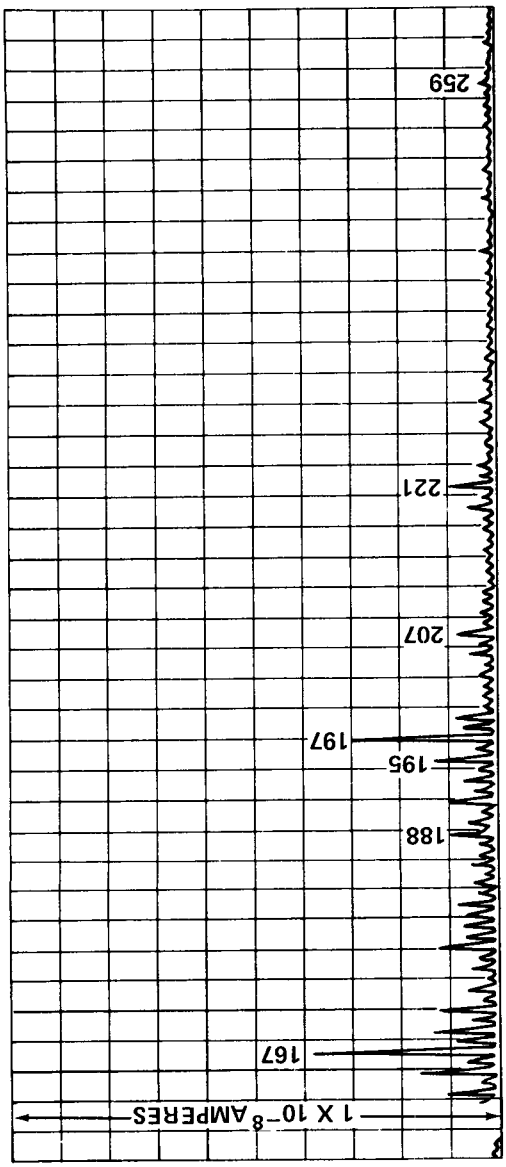
[1]Wood, G.M. and Roenick, R.J., Jr., Tables of uncertified mass spectra of diffusion pump oils, J. Vac. Sci. & Tech., (1968)

[2]Colony, J.A., Mass Spectrometry of Aerospace Materials, NASA TN D-8261 (June 1976)

[3]Bulletin 22-054a, Information About Silicone Fluids, Dow Corning Corporation, Midland, Michigan, (January 1974)



SCAN TAKEN AT TIME T = 37.25 HOURS
CHAMBER SHROUD TEMPERATURE 57° C

FIGURE 1 – RGA SCAN

4.0 FACILITY CHARACTERISTICS

The tests were conducted in Goddard Space Flight Center facility 238. The chamber is 3.7m (12 ft.) in diameter and 4.6m (15 ft.) high. An optically dense shroud provides thermal control with a capability of simulating the thermal sink of space with liquid nitrogen (LN_2). Gaseous nitrogen at $-90^{o}C(-130^{o}F)$ to $+100^{o}C(+212^{o}F)$ can be passed through the shroud for temperature control. Six 81 cm (32 in.) diffusion pumps are used to achieve hard vacuum. Each of these pumps is provided with an LN_2 trap but no isolation valve separates them from the test volume. A large LN_2 cooled plate was inserted in the chamber to provide additional pumping capacity during the chamber bake-out.

5.0 BAKE-OUT OPERATION

Figure 2 is a graph of information derived during the bake-out operation. One of the functions plotted is the saturation vapor pressure of DC-704 based on the shroud temperature. Continuous data was not recorded; the data points plotted are joined by straight line segments for presentation purposes. The shroud temperatures are noted near the data points.

The relationship between temperature and vapor pressure[3] is given in equation (1). (The equation is slightly modified for consistency of units within this report.)

$$P = \exp\left(30.279 - \frac{12825}{T+273}\right) \qquad (1)$$

where:

P = pressure, Pascal
T = temperature, degrees Celsius

The other data plotted is the peak height of m/e = 197 as determined by the RGA. Since the chamber temperature was $25^{o}C$ at the time of the first reading, this reading (actually 4.4×10^{-9} amperes) made after a one-hour warm-up, was taken as being equal to the vapor pressure of DC-704 at $25^{o}C$, 2.88×10^{-6} Pa.

The first three data points indicate that the partial pressure of DC-704 was decreasing with time. The fourth

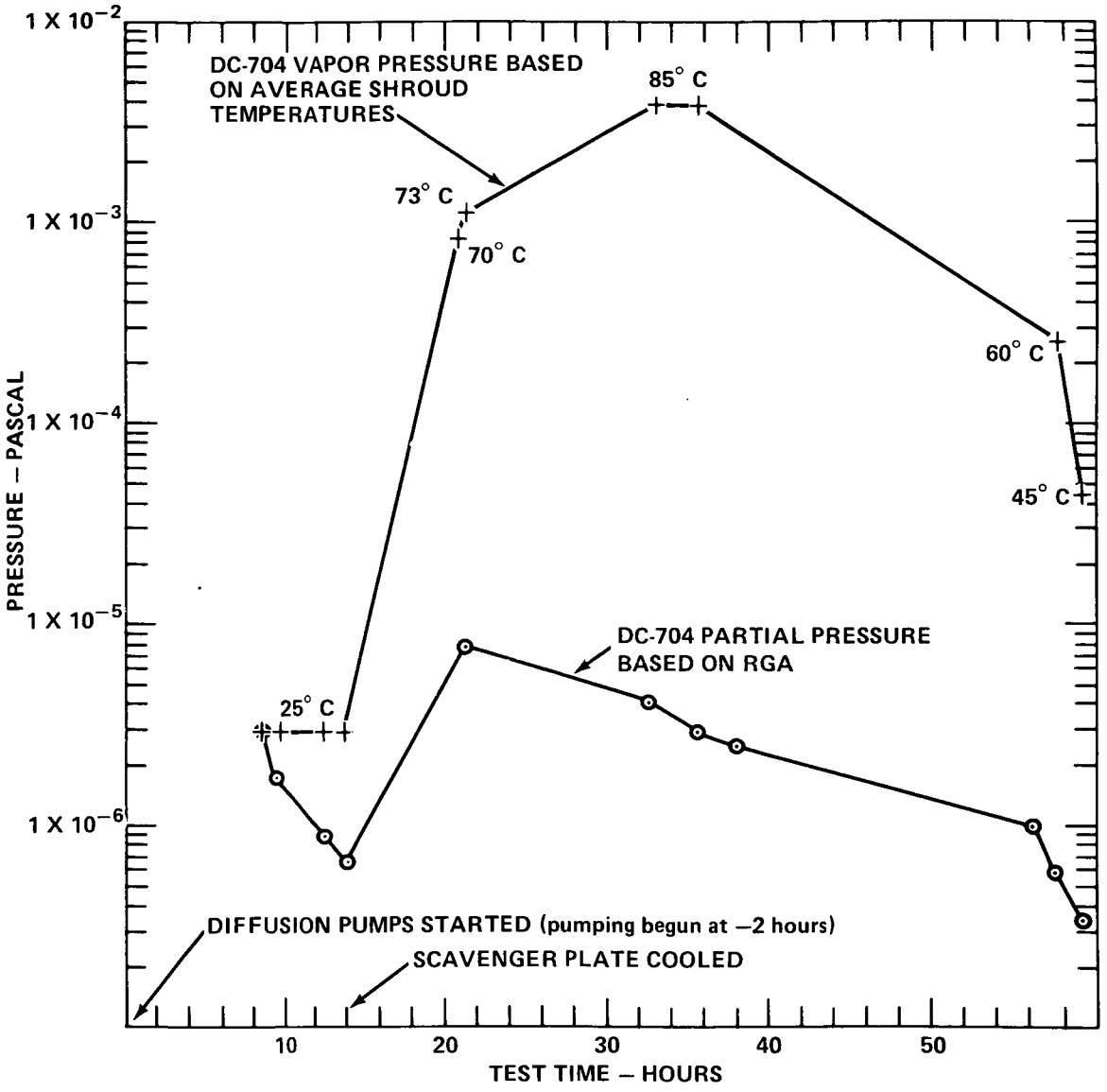


FIGURE 2 – BAKE-OUT PROFILE

data point was taken when the scavenger plate was being cooled and continues the downward trend. The points taken at 12.67 hours and 27 hours (73°C and 85°C) show that the RGA reading was decreasing in spite of an increasing wall temperature, a sign of a decreasing source since, with constant pumping speed, decreasing pressure is indicative of decreasing gas load.

The cold finger was operated at LN_2 temperatures only during the last four hours of the bake-out. The analysis indicated less than 1 mg of residue and no DC-704 was identified.

If one assumed that there was 1 mg of residue and it was all DC-704, an effective partial pressure can be estimated. One may describe the pressure in terms of temperature, flux, and molecular weight as:[4]

$$P = (T + 273)^{\frac{1}{2}} \left\{\frac{\phi}{7.78(M)^{\frac{1}{2}}}\right\} \qquad (2)$$

where:

ϕ = flux, $g/cm^2 \cdot s$
M = molecular weight, g/mole.

Substituting,

$$P = (T + 273)^{\frac{1}{2}} \left\{\frac{1 \times 10^{-3} g/(182\ cm^2 \times 4\ h \times 3600\ s/h)}{7.78\ (484)^{\frac{1}{2}}}\right\} \qquad (3)$$

Taking T = 85°C, the highest value during the entire period, a partial pressure of 4.2×10^{-8} Pa is determined. This infers a condensation temperature of less than -1°C.

In view of all the conservative assumptions and the fact that the sensitive surface was not expected to go below 0°C it was decided that a reasonable risk existed and the decision was made to proceed with the test.

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[4]Dushman, S. and Lafferty, J.M., Scientific Foundations of Vacuum Technology, John Wiley & Sons, Inc., New York (1962) p. 14

6.0 TEST OPERATIONS

Figure 3 presents data comparing the average temperature of the chamber shroud with that of the test item sensitive surface. Chamber pumpdown took place from time 0 to 5 hours at which time the pressure was 2.7×10^{-4} Pa. Return to ambient pressure took from time 308 hours (when the pressure was 2.7×10^{-5} Pa) to time 219 hours.

In Figure 4, the temperature of the test item sensitive surface has been converted to the corresponding saturation vapor pressure of DC-704; the RGA readings are in terms of partial pressure of DC-704. These transformations were accomplished as noted earlier under the section on bake-out operation. As before, a sensitivity of 655 Pa/A was used to convert the RGA electronic output to terms of pressure.

The wall temperatures were varied more slowly than usual to decrease the possibility of releasing quantities of contaminants into the chamber so rapidly that they could reach hazardous levels before the pumps could remove them.

In all cases, the partial pressure as indicated by the RGA is below the vapor pressure curve. From this we may infer that condensation did not take place. In one place, at time 37.3 hours, the RGA data point is only slightly below the vapor pressure curve. In view of the conservatism of the method, even this point was considered safe. (The minimum level of RGA capability is also noted in Figure 3.)

The cold finger analysis indicated a residue measuring 11.2 mg. DC-704 was identified. This, however, is not in itself an indication that the contamination hazard was too great. Using equation (2) and substituting, we find:

$$P = (T + 273)^{\frac{1}{2}} \left\{ \frac{11.2 \text{ mg}/(182 \text{ cm}^2 \times 303 \text{ h} \times 3600 \text{ s/h}}{7.78\ (484)^{\frac{1}{2}}} \right\} \qquad (4)$$

$$P = (T + 273)^{\frac{1}{2}} \{3.3 \times 10^{-13}\}$$

At $T = 63.5^{o}C$ (the highest average shroud temperature) the pressure based on equation (4) is 6×10^{-12} Pa, a DC-704

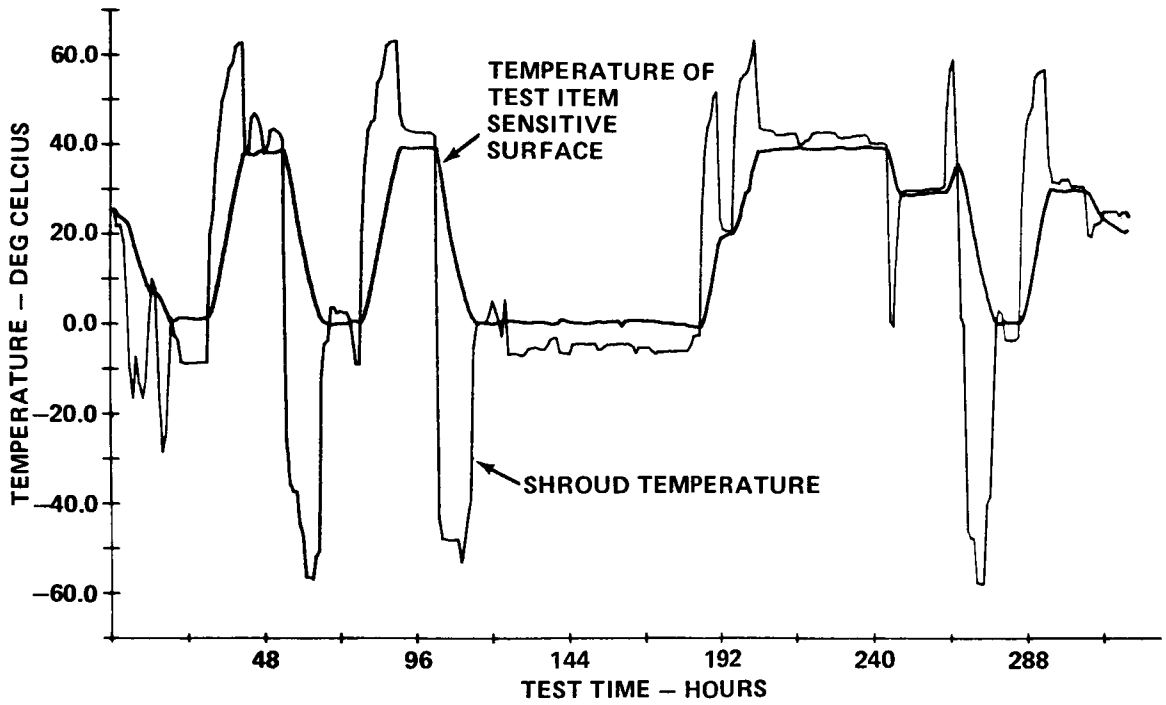

FIGURE 3 —TEST ITEM AND SHROUD TEMPERATURE PROFILE

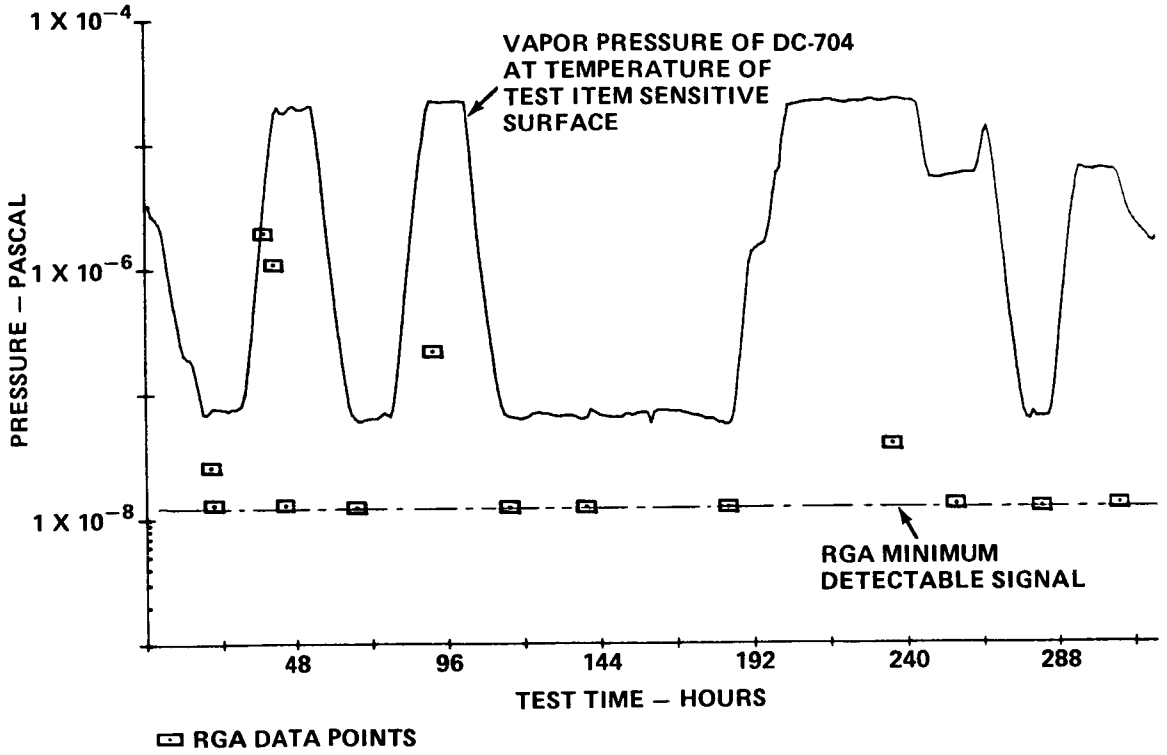

FIGURE 4 – COMPARISON OF RGA DATA WITH VAPOR PRESSURE OF DC-704 AT THE TEMPERATURE OF THE TEST ITEM SENSITIVE SURFACE.

vapor pressure which could correspond to a temperature of -44°C; no condensation on the test item is indicated.

7.0 CONCLUSIONS

The data taken during the bake-out and test operations pointed to the conclusion that the test had been conducted without condensing DC-704 diffusion pump oil on the test item. The RGA derived data indicated that the partial pressure in the chamber ambient remained below the saturation vapor pressure corresponding to the test item temperature. While the cold finger data do not provide conclusive proof that contamination did not occur, they provided no evidence that condensation would have taken place on the test item sensitive surfaces.

Most importantly, there were no test item indications that degradation had taken place because of DC-704.

8.0 RECOMMENDATIONS

The method for contaminant monitoring presented in this report depends on some knowledge of the contaminant, including identifiable peaks on an RGA scan, plus a knowledge of the contaminant vapor pressure characteristics. Based on the experience gained during this test, certain modifications to the test procedure would make the method easier and more certain in its application.

1. Maintain the RGA in a continuous scan mode. This would prevent missing data during critical periods.

2. Adjust scales on the RGA equipment so that the data may be displayed in terms of partial pressure. An additional refinement would be to process the data in real time and display it in terms of temperature corresponding to the saturation vapor pressure (taken as being equal to the RGA derived partial pressure). In this way, the temperature of the test item can be directly compared to the temperature at which condensation can occur.

3. Provide the chamber with a constant leak of a gas such as helium or neon using the partial pressure of this gas as a real time indicator of RGA performance.

MATERIAL:
300 SERIES STAINLESS STEEL

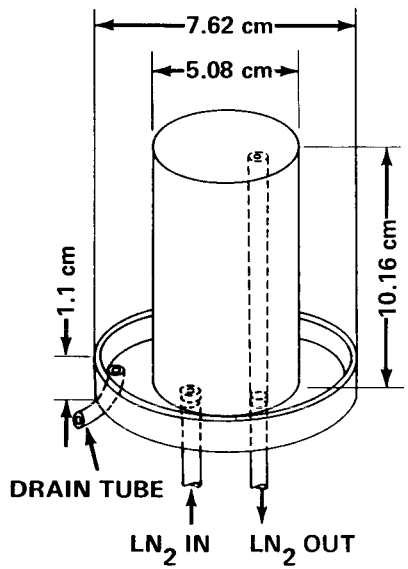

FIGURE A-1, COLD FINGER ASSEMBLY

APPENDIX A

THE COLD FINGER TECHNIQUE

The "cold finger" technique is a method used at the Goddard Space Flight Center to identify materials in the residual gases in vacuum systems. The technique employs a cold finger (see Figure A-1) which provides a surface on which these materials can deposit. The cold finger is located within the vacuum chamber test volume. The cold finger is washed with hot distilled water before the beginning of a test and then rinsed with isopropyl alcohol. The alcohol is poured over the assembly and allowed to run out through the drain tube into a container where it is retained for chemical analysis. This provides a pre-test sample.

When a test begins and the chamber is in the operating vacuum region, liquid nitrogen (LN_2) is flowed through the cold finger bringing the surface temperatures to near 80K. Species of most materials of interest condence on these cold surfaces and are retained through the course of the test. The LN_2 flow is stopped during the process of returning the vacuum chamber to room ambient and the cold finger is near room temperature when the chamber door is opened. Another alcohol rinse is obtained as quickly as possible and the sample retained for chemical analysis. It is recognized that the more volatile components are lost in this shut-down and sampling procedures, however, the process does retain much of the material which would constitute a contamination hazard.

Since approximately the same quantity of sample is taken each time (100 cm^3), a weighing of the residue provides some quantitative comparison of contaminant levels. The residue is chemically analyzed using infrared spectrophotometry plus, at times, gas-chromotography/mass-spectrometry.

If the post-test sample is shown to have low levels of contaminants, it is used as the pre-test baseline for the next test.

The total area on which material can condense and be later washed is a 234 cm^2; the area of the central cylinder (including its top) is 182 cm^2.